

# इंटरनेट

# मानक

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Mazdoor Kisan Shakti Sangathan

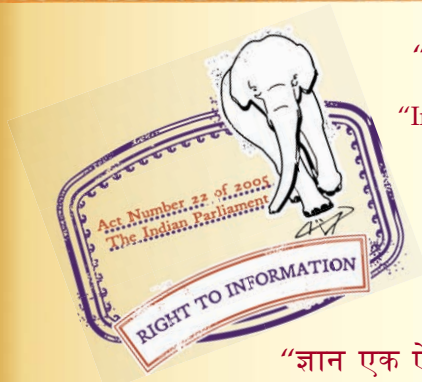
“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 330 (1992): Chromium trioxide [CHD 5: Electroplating Chemicals and Photographic Materials]



“ज्ञान से एक नये भारत का निर्माण”

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“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”



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भारतीय मानक

क्रोमियम ट्राईआक्साइड — विशिष्ट

( दूसरा पुनरीक्षण )

*Indian Standard*

CHROMIUM TRIOXIDE — SPECIFICATION

( *Second Revision* )

UDC 661 : 876 : 1

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**BUREAU OF INDIAN STANDARDS**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

*December 1992*

**Price Group 4**

## FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Electroplating Chemicals Sectional Committee had been approved by the Chemical Division Council.

In the electroplating industry chromium trioxide ( often called chromic acid ) is used in making up the chromium plating bath used for decorative coating over copper or nickel; protective coatings on all basis metals; hard deposits over ferrous metals for tools, dies and gauges; for giving an oxide film on aluminium surface; porous coatings for improved lubrication, etc.

This standard is the second revision of IS 330 : 1951 'Specification for chromic acid'. In the first revision of this standard, the chromium trioxide content was stipulated at 98.5 percent by mass as compared to that of 98.0 percent specified in the original standard. In addition, requirements of analytical grade material, previously covered in IS 559 : 1954 'Specification for chromium trioxide ( chromium acid )', analytical reagent, were also included. In this revision, special requirements for chromium trioxide for electronic industry have been added to make this standard more comprehensive.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values ( *revised* )'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Indian Standard***CHROMIUM TRIOXIDE — SPECIFICATION***( Second Revision )***1 SCOPE**

This standard prescribes the requirements and the methods of sampling and test for chromium trioxide ( chromic acid ) for use in electroplating, anodizing work and as analytical reagent.

**2 REFERENCES**

The Indian standards listed below are the necessary adjuncts to this standard:

<i>IS No.</i>	<i>Title</i>
1070 : 1992	Reagent grade water — Specification ( <i>third revision</i> )
1260 ( Part 1 ) : 1973	Pictorial markings for handling of goods: Part 1 Dangerous goods ( <i>first revision</i> )

**3 GRADES**

There shall be two grades of the material, as follows:

- Grade I — Electroplating grade, suitable for electroplating and anodizing
- Grade II — Analytical reagent grade

**4 REQUIREMENTS****4.1 Description****4.1.1 Electroplating Grade**

The material shall consist of reddish to dark brownish granules or flakes of highly hygroscopic and corrosive character.

**4.1.2 Analytical Reagent Grade**

The material shall be in the form of dark, brownish-red crystals or granular powder of highly hygroscopic character.

**4.2 Solubility**

The material of electroplating grade shall be highly soluble in water; 10 g of it when dissolved in 25 ml of water shall give a clear red solution on settling. The material of analytical

reagent grade shall be readily soluble in water, forming a clear, orange-coloured solution.

**4.3** The material shall also comply with the requirements laid down in Table 1, when tested in accordance with the method prescribed in Annex A.

**4.3.1** For use in hard chrome plating, the requirements of chromium trioxide ( as  $\text{CrO}_3$  ), matter insoluble in water and sulphate ( as  $\text{SO}_4$  ) may be as agreed to between the purchaser and the manufacturer.

**4.4 Special Requirements for Electronics Industry**

For electronics industry, the material shall also comply with the requirements given in Table 2 in addition to those given in Table 1, when tested in accordance with the methods prescribed in Annex A.

**5 PACKING**

**5.1** The material shall be packed in wide-mouth glass bottles or jars provided with well-ground air-tight glass stoppers. The electroplating grade material may also be packed in suitable air-tight steel drums.

**5.1.1** The material, when packed, shall not come in contact with organic substances, as otherwise violent action might result.

**CAUTION** — Due to its corrosive and poisonous nature, avoid contact with skin.

**6 MARKING**

**6.1** The containers, and also the packages where possible, shall be suitably marked in red letters not less than 2.5 cm high; and drums shall be marked in red letters not less than 5 cm high, with the words 'CORROSIVE, HANDLE WITH CARE'. They shall also be marked with the name, and grade of the material; its weight and indication of the source of manufacture.

**6.1.1** The packages and drums shall also be labelled as shown in Fig. 15 of IS 1260 ( Part 1 ) : 1973.

**Table 1 Requirements for Chromium Trioxide**  
( Clauses 4.3, 4.4 and 6.1.2 )

Sl No.	Characteristic	Requirements for		Method of Test
		Electroplating Grade	Analytical Reagent Grade	
(1)	(2)	(3)	(4)	(5)
i)	Chromium trioxide ( as $\text{CrO}_3$ ), percent by mass, <i>Min</i>	98.5	99.00	A-2
ii)	Matter insoluble in water, percent by mass, <i>Max</i>	0.15	To pass the test	A-3
iii)	Sulphates ( as $\text{SO}_4$ ), percent by mass, <i>Max</i>	0.20	To pass the test	A-4
iv)	Alkali salts ( as $\text{Na}_2\text{O}$ ), percent by mass, <i>Max</i>	0.30	0.200	A-5
v)	Chlorides ( as Cl ), percent by mass, <i>Max</i>	0.05	0.005	A-6
vi)	Nitrates ( as $\text{NO}_3$ ), percent by mass, <i>Max</i>	—	0.004	A-7
vii)	Aluminium, iron and trivalent chromium ( as $\text{R}_2\text{O}_3$ ), percent by mass, <i>Max</i>	—	0.030	A-8

**Table 2 Additional Requirements for Chromium Trioxide for Electronics Industry**  
( Clauses 4.4 and 6.1.2 )

Sl No.	Characteristics	Requirements	Method of Test
(1)	(2)	(3)	(4)
i)	Sulphate ( as $\text{SO}_4$ ), percent by mass, <i>Max</i>	0.005	A-4.5
ii)	Iron ( as Fe ), percent by mass, <i>Max</i>	0.01	A-9
iii)	Potassium ( as K ), percent by mass, <i>Max</i>	0.1	A-10
iv)	Sodium ( as Na ), percent by mass, <i>Max</i>	0.05	A-11

### 6.1.2 For Analytical Reagent Grade

In addition, the containers of the analytical reagent grade material shall also be labelled with full analytical data for all the characteristics of that grade prescribed in Tables 1 and 2.

### 7 SAMPLING

7.1 The method of preparing representative samples of the material and the criteria for conformity of the material to this specification shall be judged as prescribed in Annex B.

## ANNEX A

( Clause 4.3 )

### ANALYSIS OF CHROMIUM TRIOXIDE

#### A-1 QUALITY OF REAGENTS

**A-1.1** Unless specified otherwise, pure chemicals and distilled water ( *see* IS 1070 : 1992 ) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

#### A-2 DETERMINATION OF CHROMIUM TRIOXIDE

##### A-2.0 General

Chromium trioxide is determined by titrating the liberated iodine using starch solution as indicator.

## A-2.1 Reagents

### A-2.1.1 Hydrochloric Acid

Approximately 10 percent solution, prepared by diluting 270 ml of concentrated hydrochloric acid ( *see* IS 265 : 1962 ) of specific gravity 1.16 with water to 1 000 ml.

**A-2.1.2 Potassium Iodide** — solid, free from iodate.

### A-2.1.3 Standard Sodium Thiosulphate Solution

Approximately 0.1 N. Dissolve about 25 g of sodium thiosulphate crystals (  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  ) in freshly boiled distilled water, add 0.1 g of sodium carbonate ( *see* IS 296 : 1965 ) and make up the volume to 1 000 ml. Allow the solution to stand for a day and then siphon out the supernatant liquid. Pipette 25 ml of standard potassium dichromate solution ( 0.1 N ) into a conical flask. Add 5 ml of hydrochloric acid, 2 g of potassium iodide and rotate to mix. Allow to stand for 5 minutes and then add 100 ml of water. Titrate with the thiosulphate solution, using starch solution as indicator towards the end.

### A-2.1.4 Starch Solution

Triturate 1 g of pure soluble starch with 30 ml of water in a mortar. Pour the resulting paste into 1 litre of boiling water, boil for 3 minutes, allow the solution to cool and decant off the clear liquid. Prepare just before use.

## A-2.2 Procedure

Accurately weigh, out of an air-tight weighing bottle, about 5 g of the material and dissolve it in water. Make the solution up to exactly 1 000 ml and mix thoroughly. Pipette out 25 ml into a glass-stoppered flask, add 5 ml of hydrochloric acid and 3 g of potassium iodide and let it stand in the dark for 15 minutes. Dilute with 100 ml of water, and titrate the liberated iodine with standard sodium thiosulphate solution, adding starch solution towards the end of the titration and noting the end-point when the blue colour just disappears. Carry out a blank test of the reagents immediately.

## A-2.3 Calculation

$$\begin{array}{l} \text{Chromium trioxide} \\ \text{( as } \text{CrO}_3 \text{ ), per-} \\ \text{cent by mass} \end{array} = \frac{133.3 \times N \times V - V_1}{M}$$

where

$V$  = volume in millilitres of standard sodium thiosulphate solution required by 25 ml of the solution of the material,

$V_1$  = volume in millilitres of thiosulphate solution required in the blank test, and

$M$  = mass in grams of the material taken for the test

$N$  = normality of the standard sodium thiosulphate solution used

## A-3 DETERMINATION OF WATER-INSOLUBLE MATTER

### A-3.1 Procedure

Weigh accurately, out of an air-tight weighing bottle, about 20 g of the material and dissolve it in 200 ml of water. Heat on a steam bath for one hour. Filter through a tared Gooch crucible with an asbestos mat or a sintered glass crucible ( G No. 4 ). The asbestos mat on the Gooch crucible should be previously treated with a hot solution of chromic acid and dried before taring. Wash well and dry the crucible to constant weight at 105 to 110°C. Cool the crucible in a desiccator and weigh.

### A-3.2 Calculation

$$\begin{array}{l} \text{Insoluble matter,} \\ \text{percent by mass} \end{array} = \frac{100 M_1}{M}$$

where

$M_1$  = mass in grams of the residue, and

$M$  = mass in grams of the material taken for the test

### A-3.3 Determination of Insoluble Matter in Material of Analytical Reagent Grade

Weigh accurately, out of an air-tight weighing bottle, about 25 g of the material, dissolve it in water and dilute to 250 ml.

**A-3.3.1** The material shall be taken to have passed the test if the solution obtained is clear and orange coloured.

## A-4 DETERMINATION OF SULPHATES

### A-4.0 General

Sulphate are determined in the material of electroplating grade by gravimetric method and, of analytical reagent grade and electroplating grade for electronics industry by turbidity test.

### A-4.1 Reagents

**A-4.1.1 Rectified Spirit** — 95 percent by volume ( *see* IS 323 : 1959 ).

**A-4.1.2 Concentrated Hydrochloric Acid** — sp gr 1.16 ( *see* IS 265 : 1962 ).

**A-4.1.3 Barium Chloride Solution** — approximately 10 percent ( *m/v* ).

**A-4.1.4 Dilute Hydrochloric Acid** — approximately one percent ( v/v ).

**A-4.1.5 Acetic Acid** — glacial.

#### A-4.2 Procedure

Weigh accurately about 10 g of the material and dissolve in 50 ml of water. Filter into a glass beaker through a 7-cm filter paper to eliminate any insoluble matter. Wash the filter paper well with water and add to the filtrate 10 ml of rectified spirit, 35 ml of concentrated hydrochloric acid and 10 ml of glacial acetic acid. Heat until all the chromate is reduced to the green trivalent form. Boil off most of the aldehyde formed, and add to the solution 6 to 8 ml of barium chloride solution through the same filter paper. Dilute the solution to 150 ml with water, heat the solution without boiling for 4 hours and allow to stand overnight. Filter the solution containing barium sulphate through a tared sintered glass crucible ( G No. 4 ) or a Gooch crucible. Wash the barium sulphate residue with dilute hydrochloric acid till the residue is free from chromium salts and then with water till it is free from chlorides. Dry the residue to constant weight at 105° to 110°C, and weigh. Carry out a blank test on reagents immediately.

#### NOTES

1 After standing overnight filter again if there is any precipitate.

2 Excess of barium chloride is necessary to reduce the solubility of barium sulphate. Precipitation in hot solution by addition of barium chloride in a slow stream with stirring minimizes mechanical occlusion of barium chloride and gives a coarse precipitate which is less soluble in acids.

#### A-4.3 Calculation

$$\frac{\text{Sulphates ( as SO}_4 \text{ ),}}{\text{percent by mass}} = \frac{41.15 M_1}{M}$$

where

$M_1$  = mass in grams of the barium sulphate residue, and

$M$  = mass in grams of the material taken for the test.

#### A-4.4 Turbidity Test for Sulphates in Material of Analytical Reagent Grade

##### A-4.4.1 Reagents

**A-4.4.1.1 Dilute hydrochloric acid** — approximately 5 N.

**A-4.4.1.2 Barium chloride solution** — approximately 12 percent ( m/v ).

##### A-4.4.2 Procedure

Dissolve 4.00 g of the material in 50 ml of water, add 1 ml of dilute hydrochloric acid and 1 ml of barium chloride solution. Allow to stand for 3 hours.

**A-4.4.2.1** The material shall be taken to have satisfied the requirement of the test if no turbidity or a precipitate is produced in 3 hours.

#### A-4.5 Turbidity Test for Sulphates in Material of Analytical Reagent Grade

##### A-4.5.0 General

Sulphates are determined by comparing the turbidity produced by the material with barium chloride solution against that produced by a standard sulphate solution.

##### A-4.5.1 Reagents

**A-4.5.1.1 Dilute hydrochloric acid** — approximately 5 N.

**A-4.5.1.2 Barium chloride solution** — approximately 10 percent.

##### A-4.5.1.3 Standard sulphate solution

Dissolve 0.1814 g of potassium sulphate in water and make up the volume to 1000 ml. Take 10 ml of this solution and dilute it further to 100 ml. One millilitre of the final solution is equivalent to 0.01 mg of sulphate ( as  $\text{SO}_4$  ).

##### A-4.5.2 Procedure

Dissolve 4 g of the material, accurately weighed in 50 ml of water in a Nessler Cylinder. To this solution, add 1 ml of dilute hydrochloric acid and 2 ml of barium chloride solution. Stir the mixture and allow to stand for 20 minutes. Carry out a control test in the other Nessler cylinder using 2 ml of standard sulphate solution and the same quantities of other reagents, finally diluting to the mark.

**A-4.5.2.1** The limit shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

#### A-5 DETERMINATION OF ALKALI SALTS

##### A-5.1 Procedure

Carefully ignite in a porcelain dish, about 1.5 g of the material accurately weighed out of an air-tight weighing bottle, and triturate with 10 ml of hot water. Filter through a small filter paper and wash with 10 ml of hot water. Evaporate the extract. Warm the residue with 30 ml

of water, filter, wash with 10 ml of hot water and evaporate in the same dish. Ignite the evaporated residue to constant weight.

### A-5.2 Calculation

$$\text{Alkali salts ( as Na}_2\text{O ) , percent by mass} = \frac{100 M_1}{M}$$

where

$M_1$  = mass in grams of the residue, and

$M$  = mass in grams of the material taken for the test.

## A-6 DETERMINATION OF CHLORIDES

### A-6.0 General

Chlorides are determined in the material of electroplating grade by gravimetric method and, of analytical reagent grade by turbidity test.

### A-6.1 Reagents

**A-6.1.1 Concentrated Nitric Acid** — sp gr 1.42 ( see IS 264 : 1950 ).

**A-6.1.2 Silver Nitrate Solution** — approximately 5 percent ( *m/v* ).

**A-6.1.3 Dilute Ammonium Hydroxide Solution** — approximately 4 N.

**A-6.1.4 Dilute Nitric Acid** — approximately 4 N.

### A-6.2 Procedure

Weigh accurately about 10 g of the material and dissolve in 150 ml of water. Filter to obtain a clear solution. Wash the filter paper carefully and collect the washing in the beaker containing the filtrate. Make up the resulting solution to about 200 ml. Add a few drops of concentrated nitric acid and then an excess of silver nitrate solution. Allow the solution to stand overnight. Filter the solution through a Gooch crucible or sintered glass crucible ( G No. 4 ). Wash the precipitate, first with dilute nitric acid to dissolve any adhering silver chromate and then with cold water. Dissolve the precipitate in dilute ammonium hydroxide solution and reprecipitate by the addition of dilute nitric acid. Filter through a tared Gooch crucible or sintered glass crucible ( G No. 4 ) and wash with water. Dry the crucible and its contents to constant weight at  $130^\circ \pm 2^\circ\text{C}$ .

### A-6.3 Calculation

$$\text{Chlorides ( as Cl ) percent by mass} = \frac{24.74 M_1}{M}$$

where

$M_1$  = mass in grams of silver chloride, and

$M$  = mass in grams of the material taken for the test.

## A-6.4 Turbidity Test for Chlorides in Material of Analytical Reagent Grade

### A-6.4.1 Reagents

**A-6.4.1.1 Dilute nitric acid** — approximately 5 N.

**A-6.4.1.2 Silver nitrate solution** — approximately 5 percent ( *m/v* ).

### A-6.4.2 Procedure

Dissolve 1.000 g of the material in 20 ml of water and add 10 ml of dilute nitric acid. Heat the solution to  $50^\circ\text{C}$  and add 1 ml of silver nitrate solution.

**A-6.4.2.1** The material shall be taken to have not exceeded the limit prescribed in Table 1 if no turbidity is produced within 2 minutes.

## A-7 TEST FOR NITRATES

### A-7.1 Reagents

**A-7.1.1 Dilute Sulphuric Acid** — approximately 5 N.

**A-7.1.2 Hydrochloric Acid** — approximately 11 N.

**A-7.1.3 Dilute Ammonium Hydroxide Solution** — approximately 5 N.

**A-7.1.4 Barium Chloride Solution** — Dissolve 3 g of barium chloride crystals in 10 ml of water and filter.

### A-7.1.5 Standard Potassium Nitrate Solution

Dissolve 1.63 g of potassium nitrate in water and make up the volume to 1 000 ml. Take 10 ml of this solution and dilute again to 1 000 ml immediately before use; 1 ml of this solution is equivalent to 0.01 mg of nitrate ( as  $\text{NO}_3$  ).

### A-7.1.6 Standard Indigo Carmine Solution

Dissolve 0.20 g of indigo carmine in 500 ml of dilute sulphuric acid, add 20 ml of hydrochloric acid and sufficient dilute sulphuric acid to produce 1 000 ml. Standardize the solution so that 10 ml added to 2.0 ml of standard potassium nitrate solution is just decolourized on adding 12 ml of concentrated sulphuric acid and heating to boiling. One millilitre of the solution is equivalent to 0.02 mg of nitrate ( as  $\text{NO}_3$  ).

**A-7.1.7 Concentrated Sulphuric Acid** — sp gr 1.84 ( see IS 266 : 1977 ).

## A-7.2 Procedure

Weigh accurately 1 g of the material and dissolve in 5 ml of water. Add 6 ml of dilute ammonium hydroxide solution, heat to boiling, add barium chloride solution and filter. To 10 ml of the filtrate, add 1 ml of standard indigo carmine solution and 10 ml sulphuric acid and heat to boiling.

**A-7.2.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if the blue colour does not entirely disappear.

## A-8 DETERMINATION OF ALUMINIUM, IRON AND TRIVALENT CHROMIUM

### A-8.1 Reagents

**A-8.1.1 Ammonium Chloride** — solid.

**A-8.1.2 Ammonium Hydroxide Solution** — sp gr 0.90.

**A-8.1.3 Ammonium Nitrate Solution** — approximately 1 percent ( *m/v* ).

**A-8.1.4 Dilute Hydrochloric Acid** — 1:1 ( *v/v* ).

**A-8.1.5 Methyl Red Indicator Solution** — prepared by dissolving 0.15 g in 500 ml of water.

### A-8.2 Procedure

Weigh accurately about 5 g of the material and dissolve in 100 ml of water. Heat to boiling, add 1 g of ammonium chloride, continue boiling till the solution is clear and then add ammonium hydroxide solution till the solution is distinctly alkaline to methyl red solution. Boil off excess of ammonia and filter through a filter paper ( Whatman No. 31 or 41 ). After washing the precipitate on the filter paper several times with ammonium nitrate solution, dissolve it in minimum quantity of dilute hydrochloric acid, add 1 g of ammonium chloride and repeat the precipitation as above. Boil the liquor with the precipitate and filter it through a filter paper ( Whatman No. 31 or 41 ), wash the precipitate thoroughly with ammonium nitrate solution and then with water till the washings are free from chlorides. Ignite the precipitate in a tared crucible to constant weight.

### A-8.3 Calculation

$$\begin{array}{l} \text{Aluminium, iron and trivalent} \\ \text{chromium ( as } R_2O_3 \text{ ),} \\ \text{percent by mass} \end{array} = \frac{100 M_1}{M}$$

where

$M_1$  = mass in grams of the residue, and

$M$  = mass in grams of the material taken for the test.

## A-9 DETERMINATION OF IRON ( AS Fe )

### A-9.1 Reagents

**A-9.1.1 Concentrated Nitric Acid** — See IS 264 : 1976.

**A-9.1.2 Ammonium Persulphate** — solid.

**A-9.1.3 Butanolic Potassium Thiocyanate** — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient n-butanol to make up to 100 ml and shake vigorously until the solution is clear.

**A-9.1.4 Dilute Sulphuric Acid** — Approximately 10 percent ( *v/v* ).

**A-9.1.5 Standard Iron Solution**

Weigh 0.702 g of ferrous ammonium sulphate [  $FeSO_4(NH_4)_2SO_4 \cdot 6H_2O$  ] and dissolve in 10 ml of dilute sulphuric acid.

Dilute with water to make up the volume to 1 000 ml. Transfer 10 ml of this solution to a 100 ml volumetric flask and again dilute with water to make up the volume to 100 ml. One millilitre of this solution is equivalent to 0.01 mg of iron ( as Fe ).

### A-9.2 Procedure

Take about 0.5 g of the sample accurately, weighed, dissolve it in 10 ml water. Add 1 ml of concentrated nitric acid and boil. Cool, transfer the solution to a Nessler cylinder and add 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Shake for 30 seconds and allow the liquid to separate. Carry out a control test in the other Nessler cylinder, adding slowly from a burette a quantity of the standard iron solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture until the colours of butanol layers in the two cylinders are exactly matched.

### A-9.3 Calculations

$$\begin{array}{l} \text{Iron ( as Fe ),} \\ \text{percent by mass} \end{array} = \frac{V}{M} \times 0.001$$

where

$V$  = Volume in ml of standard iron solution required in the control test, and

$M$  = Mass in g of the material present in the aliquot of prepared sample solution.

## A-10 DETERMINATION OF POTASSIUM (AS K)

### A-10.1 Reagents

**A-10.1.1 Rectified Spirit** — See IS 323 : 1959.

**A-10.1.2 Prechloric Acid** — 40 to 60 percent.

**A-10.1.3 Concentrated Hydrochloric Acid** — See IS 265 : 1976.

**A-10.1.4 Phenolphthalein Indicator Solution** — Dissolve 0.1 g of the material in 100 ml of 60 percent rectified spirit.

**A-10.1.5 Acetic Acid** — glacial (See IS 695 : 1986) and 20 percent solution.

### A-10.1.6 Sodium Cobaltinitrite Solution

Dissolve 50 g of sodium cobaltinitrite and 25 g of sodium acetate in 250 ml of water. Allow the solution to stand for 24 hours and then filter.

### A-10.2 Procedure

**A-10.2.1** Dissolve about 5 g of the material in 10 ml of water. Add 0.5 ml of 20 percent acetic acid and drop by drop, while stirring, 5 ml of the sodium cobaltinitrite solution and allow to stand for several hours, preferably overnight. Then filter through a filter crucible, previously washed with cold water containing a little

reagent and dried at  $105 \pm 2^\circ\text{C}$  to constant mass and weighed to the nearest 0.000 2 g.

**A-10.2.2** Wash the residue on the filter three times with 20 percent acetic acid using 5 ml each time, and twice with rectified spirit, using 5 ml each time, dry at a temperature of  $105 \pm 2^\circ\text{C}$  to constant mass, cool in a desiccator and weigh to the nearest 0.000 2 g. One gram of a precipitate of the composition  $\text{K}_2\text{NaCo}(\text{NO}_2)_6 \cdot \text{H}_2\text{O}$  is assumed to contain 0.172 2 g of potassium. It is preferable to determine this factor, taking only small quantities of a potassium salt.

### A-10.3 Calculation

$$\text{Potassium (as K), percent by mass} = \frac{M_1 \times 0.172\ 2 \times 100}{M}$$

where

$M_1$  = mass in g of the dried residue, and

$M$  = mass in g of the material taken for the test.

**A-10.4** Alternatively determine potassium by flame photometer at  $766.5\ \text{m}\mu$  according to the directions of the manufacturer of the apparatus.

## A-11 DETERMINATION OF SODIUM (AS Na)

**A-11.1** Determine sodium by flame photometer at  $589\ \text{m}\mu$  according to directions of the manufacturer of the apparatus.

## ANNEX B

( Clause 7.1 )

### SAMPLING OF CHROMIUM TRIOXIDE

#### B-1 GENERAL REQUIREMENTS OF SAMPLING

**B-1.0** In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

**B-1.1** Samples shall be taken in a protected place not exposed to damp air, dust or soot.

**B-1.2** The sampling instrument shall be clean and dry.

**B-1.3** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**B-1.4** To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

**B-1.5** The samples shall be placed in suitable, clean, dry and air-tight glassbottles or other suitable containers on which the material has no action.

**B-1.6** The sample containers shall be of such a size that they are almost completely filled by the sample.

**B-1.7** Each sample containers shall be sealed air-tight after filling, and marked with full details of sampling, and the date of sampling.

**B-2 SCALE OF SAMPLING****B-2.1 Lot**

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute the lot. If a consignment is declared to consist of different batches of manufacturer, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

**B-2.2** For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately. The number of containers to be selected at random from lots of different sizes shall be as given below:

<i>Lot Size ( N )</i>	<i>Sample Size ( n )</i>
3 to 15	3
16 „ 40	4
41 „ 110	5
111 „ 180	6
181 „ 300	7
301 „ 500	8
501 „ 800	9
801 and above	10

**B-2.3** In order to ensure randomness of selection, random number tables shall be used. If such tables are not available, the following procedure is recommended for use:

Arrange all the containers in the lot in a systematic manner and starting from any one, count them as 1, 2, 3,....., up to  $r$  so on and where  $r$  is the integral part of  $N/n$  ( see **B-2.2** ). Every  $r$ th container thus counted shall be withdrawn to constitute the sample.

**B-3 INDIVIDUAL SAMPLES AND COMPOSITE SAMPLES**

**B-3.1** From each of the containers selected according to **B-2.3**, a representative portion of the material sufficient for carrying out the tests,

specified under 4, shall be drawn and this shall constitute the individual sample.

**B-3.2** From each of the individual samples, a small but equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample.

**B-3.3** The material constituting the individual and composite samples shall be transferred to separate bottles and labelled with full identification particulars of the samples.

**B-4 NUMBER OF TESTS**

**B-4.1** Tests for the determination of chromium trioxide content shall be carried out on each of the individual samples.

**B-4.2** Tests for the remaining characteristics shall be done on the composite sample.

**B-5 CRITERIA FOR CONFORMITY****B-5.1 For Individual Samples**

For chromium trioxide content, the mean and range of test results shall be computed as follows:

$$\text{Mean ( } \bar{X} \text{ )} = \frac{\text{sum of individual test results}}{\text{number of tests}}$$

$$\text{Range ( } R \text{ )} = \text{difference between the maximum and the minimum values of the test results.}$$

**B-5.1.1** The lot shall be declared to have satisfied the requirements for chromium trioxide content if  $\bar{X} - 0.6 R \geq 98.5$  for Electroplating Grade and  $\bar{X} - 0.6 R \geq 99.00$  for Analytical Reagent Grade and for electroplating grade for electronics industry respectively.

**B-5.2 For Composite Sample**

For declaring the conformity of the lot to the requirements of all other characteristics ( of both the grades ), the test results on the composite sample shall meet the corresponding specified requirements.

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